

# PATENT SPECIFICATION

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## (54) SOLID SOAPS

(71) We, ETHYL CORPORATION, a Corporation organised under the laws of the State of Virginia, United States of America, of 330 South Fourth Street, Richmond, State of Virginia, United States of America and KAO SOAP COMPANY LIMITED, a Japanese Body Corporate, of Tokyo, Japan, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to solid soaps, such as soaps in bar form for skin or fabric washing where controlled solubility and foaming properties, as well as absence of skin irritation, are important aspects.

As is well known soaps can be manufactured by the reaction of fatty acids or their esters with caustic alkali to produce metal salts of the organic acids employed in the starting material. For economic and other reasons, it has been conventional in batch or continuous saponification to use a mixed fatty acid system based on natural oils and fats; in particular, fatty acids or esters of animal tallow or fats such as beef and fatty acids or esters of plant oils such as coconut oil. Such acids, oils, fats and esters consist essentially of straight carbon chain molecules. In general, the beef tallow source acids or esters contain a considerable percentage of unsaturate molecules, about 50 percent by weight, including 5 percent or more by weight of polyunsaturated molecules such as those of the type of linoleic acid and linolenic acid. Such unsaturated materials, particularly the polyunsaturated ones, undergo comparatively rapid deterioration when the soap products containing them are stored, frequently requiring that after several months of storage, such soaps must be returned to the producer and reworked. Heretofore, however, the inclusion of unsaturated fatty acid soaps has been considered essential because of the processing and use physical properties conveyed by

such acids and soaps, in particular melting point and solubility characteristics.

According to the present invention there is provided a solid soap comprising soaps of one or more alkali metals and a mixed fatty acid system containing from 60 to 85 percent by weight of a straight chain saturated fatty acid having from 8 to 18 carbon atoms per molecule and from 15 to 40 percent by weight of a branched chain saturated fatty acid having from 10 to 24 carbon atoms per molecule, and having a titre of from 35 to 45°C, from 7 to 30 percent by weight based on the soaps of a straight chain substantially saturated free fatty acid having from 8 to 18 carbon atoms per molecule, and an inorganic salt of a strong base and a strong acid in an amount from 0.4 to 3.0 percent by weight based on the soaps.

The invention also provides a process for producing a solid soap, which process comprises saponifying a mixed fatty acid system containing from 60 to 85 percent by weight of a straight chain saturated fatty acid having from 8 to 18 carbon atoms per molecule and from 15 to 40 percent by weight of a branched chain saturated fatty acid having from 10 to 24 carbon atoms per molecule, or their esters, and having a titre of from 35 to 45°C, with about a neutralising amount of a caustic alkali to form a neat soap, and then adding as a superfatting agent from 5 to 20 percent by weight based on the neat soap of a straight chain substantially saturated fatty acid having from 8 to 18 carbon atoms per molecule, there being added to the foregoing mixture or to at least one of the components thereof an inorganic salt in an amount of from 0.3 to 2 percent by weight based on the neat soap.

Using the foregoing components, soaps are produced having excellent properties as regards stability against deterioration, ability to foam readily at low temperatures and ability to disperse scum. These soaps avoid abnormal processing problems so that bar

forming operations can be similar to those used with prior art soaps.

In the process of the invention it is preferred for convenience to saponify free acids, but if desired all or part of the acids may be replaced by corresponding esters of alcohols, typically monobasic alcohols such as alkanols, or polybasic alcohols, typically dibasic alcohols such as glycols or higher polybasic alcohols such as glycerols. Thus methyl esters or natural fats and oils (glycerol based esters) are suitable substitutes.

Such a mixed system is typically saponified by treatment with an aqueous system containing a neutralising amount of caustic alkali such as hydroxide or carbonate of one or more elements of the alkali or alkaline earth groups of the Periodic Table (Fisher Scientific Company, 1955). The aqueous system may be typically about 30 percent by weight of caustic and about 70 percent water. For low melting point properties, a combination of sodium and potassium is preferred such as from 10 to 30 mol percent potassium hydroxide and from 70 to 90 mol percent sodium hydroxide. (If the amount of the caustic potash is made more than 30 percent, the produced soap will be too soft but, if it is made less than 10 percent, the foaming ability at low temperature is impaired). Preferred caustic alkalies are those of sodium or potassium or mixtures thereof.

The amount of water used in the aqueous saponification is preferably adjusted to produce a final neat soap containing about 30 percent water by weight. This adjustment is made by using aqueous caustic alkali solutions of the proper initial water content, optionally supplemented with other conventional moisture alteration manipulations such as adjustment of the duration of boiling in batch processes or the addition of water.

The saponification is preferably conducted to completion with negligible excess acid or caustic alkali remaining. Where esters are used as the source of part or all of acid, the by-product alcohol is removed in conventional manner which may include salting out, decantation or filtering. The saponification processing of fatty acids or their esters is well known in the prior art as discussed, for example, in Encyclopedia of Chemical Technology, 2nd Edition, Volume 18, Kirk-Othmer, Interscience, New York, 1969, pp. 415-432.

The neat soap from the foregoing is then combined with from 5 to 20, preferably 8 to 15, percent by weight based on the total amount of the neat soap of a saturated fatty acid having from 8 to 18 carbon atoms per molecule, preferably essentially all molecules being straight chain, as a superfatting

agent. (This is 7 to 30 percent by weight based on the water free soap content). The superfatting agent imparts improved properties to the soap. Although the amount of superfatting agent is specified on a basis of neutralised neat soap without excess caustic, the proportions are subject to further variation if desired, to accommodate for variation of the neat soap from a neutralised condition. The superfatted neat soap generally also contains from 0.3 to 2.0 percent by weight based on the neat soap (0.4 to about 3.0 percent by weight based on the water free soap content) of an inorganic salt such as sodium chloride or potassium chloride. Although salting-out is frequently used in the separation of glycerol or other ester by-product, this particular quantity of salt content is salt in the soap and is included even where acids rather than esters are employed. When acids are used, the point in the process where the salt addition occurs is not critical. For example, the salt can be added to the aqueous caustic alkali system or added to the soap before or after the addition of the superfatting acid. With esters this contained salt is generally added, at least in part, after the salting-out operations.

Typical straight chain saturated fatty acids which may be used in the present invention are caprylic acid, capric acid, lauric acid, myristic acid, palmitic acid, stearic acid, and mixtures thereof. Such acids are typically obtained from beef tallow and coconut oil and other natural oils and fats. In some instances, hydrogenation purification of such natural materials is desired to reduce unsaturation. Additionally, acids produced synthetically such as by caustic fusion of alcohols or by oxidation of hydrocarbons are also suitable.

Typical branched chain fatty acids which may be used have the general formula:



wherein  $R_2$  is an alkyl group having up to four carbon atoms such as methyl, ethyl, propyl or butyl;  $R_1$  is an alkyl group having from 4 to 21 carbon atoms and the total number of carbon atoms of  $R_1$  and  $R_2$  is from 8 to 22. Specific acids are: 2-butyl-caproic acid; 2-butyl- or 2-propyl-enanthic acid; 2-butyl-, 2-propyl- or 2-ethylcaprylic acid; 2-methyl-, 2-ethyl-, 2-propyl- or 2-butyl-perlargononic acid; similarly branched capric acid, undecanoic acid, lauric acid, tridecanoic acid, myristic acid, pentadecanoic acid, palmitic acid, margaric acid, stearic acid, nonadecanoic acid, arachic acid; 2-propyl-, 2-ethyl- or 2-methyl-heneicosanic acid; 2-ethyl- or 2-methyl-behenic acid; 2-

methyltricosanic acid; and mixtures thereof.

The straight chain saturated fatty acid and branched chain fatty acid (or ester equivalents of either or both) are mixed in a weight ratio of from 60:40 to 85:15, the mixing ratio being adjusted to provide a titre of the mixed acids which is from 35 to 45°C. The average number of carbon atoms per molecule of all the fatty acids or acid components in such mixed systems is from 12 to 18. The number of carbon atoms in the raw material fatty acids or esters is limited to those specified not only by the requirement for the titre of the mixed fatty acid but also by other factors such as skin irritation and solubility connected with the fabrication and use of the product soap.

Where the raw material fatty acids are used in the form of an ester mixture, it is desirable that the composition and titre of fatty acids corresponding to the acid components of the esters fall in the above mentioned ranges.

The scum dispersibility of the soaps of the invention may be improved further by blending with a non-ionic surface active agent. Suitable surface active agents are fatty acid alkanol amides, e.g. alkanol or dialkanol amides, of a fatty acid having from 8 to 18, e.g. 10 to 18, carbon atoms per molecule. Preferably the alkanol amide is an ethanolamide. These alkanol amides may usefully be employed as their adducts with alkylene oxides, e.g. having an average of up to 10 alkylene oxide units per molecule. Preferred adducts are those with ethylene oxide. The amount of such non-ionic surface active agent is preferably from 5 to 15 percent by weight based on the total amount of the neat soap, or about 7 to 22 percent by weight on an anhydrous basis.

Following is a description by way of example of the production of solid soaps in accordance with the invention.

#### Example 1

A first acid mixture containing saturated branched chain and straight chain fatty acids was produced from oxo process alcohols. The branched chain acids had mostly methyl and butyl radicals attached to the carbon atom in the alpha position relative to the carboxyl group.

The acid composition in weight percent was as follows:

| <i>Acid</i>                       |                            |                                  |
|-----------------------------------|----------------------------|----------------------------------|
| <i>(Carbon Atom per Molecule)</i> | <i>Branched Fatty Acid</i> | <i>Straight Chain Fatty Acid</i> |
| C <sub>12</sub>                   | 8.9                        | 11.8                             |
| C <sub>13</sub>                   | 16.9                       | 16.3                             |
| C <sub>15</sub>                   | 13.9                       | 13.8                             |
| C <sub>17</sub>                   | 9.8                        | 8.6                              |
| 65                                | 49.5                       | 50.5                             |

The acids analysed as follows:

|                      |  |
|----------------------|--|
| Saponification value | 249.7 (milligrams KOH per gram of sample)                    |
| Acid value           | 248.1 (milligrams KOH per gram of sample)                    |
| Iodine value         | 0.2 (centigrams iodine (I <sub>2</sub> ) per gram of sample) |
| Titre                | 20.6°C. (melting point)                                      |

A second acid mixture containing saturated straight chain fatty acids was prepared by combining 70 parts by weight of industrial stearic acid obtained by fractional distillation of mixed natural source fatty acid and 30 parts by weight of natural source industrial palmitic acid containing 1.5 percent of palmitoleic acid and oleic acid. The titre of the mixture was 54.3°C.

The mixture composition was as follows:

| <i>Acid</i>                        | <i>Weight percent</i> |
|------------------------------------|-----------------------|
| <i>(Carbon Atoms per Molecule)</i> |                       |
| C <sub>10</sub>                    | 0.1                   |
| C <sub>12</sub>                    | 0.6                   |
| C <sub>14</sub>                    | 3.6                   |
| C <sub>15</sub>                    | 0.5                   |
| C <sub>16</sub>                    | 46.6                  |
| C <sub>17</sub>                    | 2.1                   |
| C <sub>18</sub>                    | 42.2                  |
| Unknown acid                       | 2.8                   |
| Miscellaneous                      | 1.5                   |
|                                    | 100.0                 |

The above mentioned first acid and second acid mixtures were combined in various proportions so as to provide a series of fatty acid compositions. In turn, each of the mixtures was put into an open saponifier. To each was added a aqueous solution of about 28 percent by weight sodium hydroxide containing table salt (sodium chloride) in an amount to provide 0.3 percent by weight on the entire neat soap system after the saponification. The caustic was added to the heated fatty acid gradually while it was boiled and neutralised. At a point where no more caustic alkali was consumed, the neutralisation was stopped providing a soap containing less than 0.05 percent of free alkali and about 30 percent water. The fatty acid superfatting agent and other additives were then added in various proportions as indicated in subsequent tabulations.

Solid soap bars were then made using a conventional mechanical kneading and moulding processing. In this process the neat soap was solidified, cut into small pieces, dried, and kneaded. This firm soap was then extruded and cut into sections which were molded into bars using a plain (non-decorative) die.

Samples for the evaluation of soap

properties of various mixtures were taken from the solid soap tabulated hereinafter.

The workability was determined by the observation of the state of the soap in the 5 aforementioned kneading and extruding steps.

The methods of testing the characteristics mentioned in the table shown hereinafter were as follows:

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#### 1) Solubility by abrasion.

Testing according to a standard J.I.S. procedure (Japanese Industrial Standard K-3304—1956, Section 4.7).

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In this test a sample of prescribed surface area was placed in contact with a continuous moving belt dipping in water under prescribed conditions of time, speed, tem-

perature, pressure, etc. The weight loss during the test per unit of surface area contacting the belt provides a comparative abrasion test result. 20

#### 2) Dipping Test

##### a) Swelling rate

A piece of soap 1×1×5 cm. was immersed in water at 25°C. for 4 hours. The percentage of weight increase due to the absorption of water was designated swelling rate. 25

##### b) Solubility

The weighed sample after the above test 2-(a) was dried at 105°C. for 4 hours and the weight of the anhydrous soap remaining was determined. 30

(Weight of the measured value of anhydrous soap as converted to the weight of original hydrous soap)

40

$$\text{Solubility (\%)} = \frac{(\text{Weight of soap before dipping}) - (\text{Weight of soap after dipping})}{(\text{Weight of soap before dipping})} \times 100$$

#### 45 c) Cracking Degree

The surface state of the sample from (2-b) above (after dipping in water at 25°C. for 4 hours and wind-drying at room temperature for 24 hours). The state was judged 50 visually according to standards which rated the original form as 0 and a remarkable or severe cracking as 5.

be at a fixed constant temperature and the number of strokes of the sponge required to cover the surface of water in the vessel with foam was recorded. This test was made according to the method shown by Paul Bessher on pages 53 to 57 in the Journal of American Oil Chemists Society, Volume 34, 1957. 70

#### 3) Melting Collapse

55 A piece of soap of 2.5×5.0×1.2 cm. (having a surface area of about 40 cm<sup>2</sup>) was prepared and was immersed in 80 cc of water at 30°C. for 4 hours. The swollen part was rubbed off and the reduction of 60 the weight thereby was expressed in g/100 cm<sup>2</sup> of surface area.

#### 5) Consumption Degree

This was tested as an extension of the test in (4) aforementioned. The weight loss in grams after a fixed number of strokes was shown. 80

Mixture 15 in the following tabulation is prior art commercial sodium salt toilet soap as is made from beef tallow and coconut oil in about 80/20 weight ratio of acids of the two types containing about 40 percent unsaturated soaps. 85

#### 4) Quick Foamability

65 A soap bar with a surface of 2.5 cm. × 5.0 cm. was uniformly rubbed on that surface with a rubber sponge under fixed conditions using 6 litres of water adjusted to

Added fatty acid (2) in the following tabulation represents excess fat agents having the following characteristic values: 90

|    |     | Sap Value        | Acid Value | Iodine Value | Titre (°C.) | C <sub>17</sub> -C <sub>19</sub> Fatty Acid |
|----|-----|------------------|------------|--------------|-------------|---|
| 95 | (a) | 260.3            | 258.5      | 5.2          | 29.3        | 95.9%                                       |
|    | (b) | (not determined) | 267.5      | 9.0          | 24.1        | 94.9%                                       |

## COMPOSITIONS

## MIXTURES 1-8

| 5 Mixture No.  | 1     | 2     | 3     | 4     | 5     | 6     | 7     | 8     |
|--|-------|-------|-------|-------|-------|-------|-------|-------|
| <i>Mixture:</i><br>(weight parts)                    |       |       |       |       |       |       |       |       |
| 10 Branched chain/straight chain fatty acid mixture. | 70    | 65    | 60    | 44    | 44    | 44    | 55    | 55    |
| Straight chain fatty acid mixture.                   | 30    | 35    | 40    | 56    | 56    | 56    | 45    | 45    |
| <i>15 Composition:</i><br>(Total)                    |       |       |       |       |       |       |       |       |
| Branched fatty acid % wt.                            | 32.5  | 30.2  | 27.8  | 20.4  | 20.4  | 20.4  | 25.5  | 25.5  |
| 20 Straight chain saturated fat acid in %            | 66.2  | 68.3  | 70.4  | 77.2  | 77.2  | 77.2  | 72.5  | 72.5  |
| Straight chain unsaturated fatty acid in %           | 0.5   | 0.5   | 0.6   | 0.8   | 0.8   | 0.8   | 0.7   | 0.7   |
| Unknown acid in %                                    | 0.8   | 1.0   | 1.2   | 1.6   | 1.6   | 1.6   | 1.3   | 1.3   |
| Average titre in °C                                  | 35.7  | 37.9  | 39.7  | 44.3  | 44.3  | 44.3  | 40.9  | 40.9  |
| 25 NaOH/KOH (mol %)                                  | 100/0 | 100/0 | 100/0 | 70/30 | 75/25 | 80/20 | 75/25 | 85/15 |
| Added fatty acid (2) (in parts by wt.)               | —     | —     | —     | —     | —     | —     | —     | —     |

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## PROPERTIES

## MIXTURES 1-8

| 35 Mixture No.   | 1           | 2           | 3        | 4     | 5     | 6    | 7     | 8     |
|--|-------------|-------------|----------|-------|-------|------|-------|-------|
| <i>Solubility by Abrasion</i>                            |             |             |          |       |       |      |       |       |
| At 10°C for 30 min.                                      | 2.1         | 2.0         | 0.6      | 20.1  | 16.1  | 13.4 | 17.4  | 19.1  |
| At 40°C for 30 min.                                      | 139.3       | 100.4       | 65.1     | 132.4 | 108.3 | 90.6 | 131.7 | 132.5 |
| 40 <i>Dipping test at 25°C for 4 hrs.</i>                |             |             |          |       |       |      |       |       |
| <i>Swelling rate</i>                                     | 81.7        | 64.8        | 38.7     | 56.6  | 52.7  | 53.8 | 63.3  | 64.5  |
| <i>Solubility</i>  | 6.5         | 5.1         | 4.3      | 19.8  | 16.5  | 15.1 | 20.8  | 17.7  |
| 45 <i>Cracking</i>                                       | 1.5         | 1.5         | 1.5      | 4.0   | 3.0   | 3.0  | 2.5   | 3.0   |
| <i>Melting Collapse in g/100 cm² at 30°C for 4 hours</i> |             |             |          |       |       |      |       |       |
|  | 4.32        | 0.57        | -0.32    | 14.9  | 14.5  | 13.8 | 13.1  | 12.8  |
| 50 <i>Quick Foamability</i>                              |             |             |          |       |       |      |       |       |
| Number of strokes at 10°C                                | 100<        | 100<        | 100<     | 10    | 12    | 12   | 10    | 12    |
| Number of strokes at 40°C                                | 23          | 23          | 41       | 9     | 10    | 10   | 10    | 11    |
| 55 <i>Consumption degree</i>                             |             |             |          |       |       |      |       |       |
| In g/100 strokes at 10°C                                 | 3.6         | 3.4         | 3.0      | 3.5   | 3.1   | 3.1  | 3.1   | 3.0   |
| 60 In g/50 strokes at 40°C                               | 3.6         | 3.4         | 3.3      | 3.0   | 2.9   | 2.8  | 3.0   | 2.9   |
| <i>Workability</i>                                       |             |             |          |       |       |      |       |       |
| 65 Subjective Opinion                                    | Rather good | Rather good | Not good | Good  | Good  | Good | Good  | Good  |

## COMPOSITIONS

## MIXTURES 9-15

| 5 Mixture No.                               | 9           | 10          | 11          | 12         | 13          | 14          | 15    |
|---|-------------|-------------|-------------|------------|-------------|-------------|-------|
| Mixture:<br>(weight parts)                  |             |             |             |            |             |             |       |
| Branched chain/straight                     |             |             |             |            |             |             |       |
| 10 chain fatty acid mixture.                | 38.7        | 38.7        | 38.7        | 40.5       | 39.5        | 37.8        | —     |
| Straight chain fatty acid mixture.          | 49.3        | 49.3        | 49.3        | 51.5       | 50.5        | 48.2        | —     |
| 15 Composition:<br>(Total)                  |             |             |             |            |             |             |       |
| Branched fatty acid in % wt.                | 20.4        | 20.4        | 20.4        | 20.4       | 20.4        | 20.4        | —     |
| 20 Straight chain saturated fatty acid in % | 77.2        | 77.2        | 77.2        | 77.2       | 77.2        | 77.2        | —     |
| Straight chain unsaturated fatty acid in %  | 0.8         | 0.8         | 0.8         | 0.8        | 0.8         | 0.8         | —     |
| Unknown acid in %                           | 1.6         | 1.6         | 1.6         | 1.6        | 1.6         | 1.6         | —     |
| Average titre in °C                         | 44.3        | 44.3        | 44.3        | 44.3       | 44.3        | 44.3        | 44    |
| 25 NaOH/KOH (mol %)                         | 70/30       | 75/25       | 80/20       | 80/20      | 80/20       | 80/20       | 100/0 |
| Added fatty acid (2)<br>(in parts by wt.)   | (a)<br>12.0 | (a)<br>12.0 | (a)<br>12.0 | (b)<br>8.0 | (b)<br>10.0 | (b)<br>14.0 | —     |

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## PROPERTIES

## MIXTURES 9-15

| 35 Mixture No.  | 9     | 10    | 11    | 12    | 13    | 14    | 15    |
|---|-------|-------|-------|-------|-------|-------|-------|
| Solubility by Abrasion  |       |       |       |       |       |       |       |
| At 10°C for 30 min.   | 30.4  | 29.1  | 27.9  | 20.5  | 21.3  | 30.7  | 10.1  |
| At 40°C for 30 min.   | 155.3 | 148.0 | 128.6 | 115.1 | 137.2 | 141.4 | 128.6 |
| 40 Dipping test at 25°C<br>for 4 hrs.                               |       |       |       |       |       |       |       |
| Swelling rate   | 39.4  | 34.3  | 33.0  | 43.8  | 41.5  | 38.2  | 53.9  |
| Solubility  | 25.0  | 23.2  | 19.6  | 17.8  | 21.3  | 17.7  | 8.2   |
| 45 Cracking   | 0     | 1     | 1.5   | 1.5   | 1.5   | 1.5   | 4     |
| Melting Collapse in<br>g/100 cm <sup>2</sup> at 30°C<br>for 4 hours | 12.2  | 10.9  | 10.1  | 10.1  | 10.0  | 10.0  | 11.9  |
| 50 Quick Foamability  |       |       |       |       |       |       |       |
| Number of strokes at 10°C   | 16    | 16    | 19    | 12    | 14    | 18    | 197   |
| Number of strokes at 40°C   | 14    | 15    | 16    | 11    | 11    | 11    | 30    |
| 55 Consumption degree   |       |       |       |       |       |       |       |
| In g/100 strokes at 10°C  | 2.67  | 2.48  | 2.09  | 2.61  | 2.57  | 2.59  | 1.02  |
| 60 In g/50 strokes at 40°C  | 2.90  | 2.79  | 2.56  | 2.94  | 2.92  | 3.16  | 2.31  |
| Workability   |       |       |       |       |       |       |       |
| Subjective Opinion  | —     | —     | —     | —     | —     | —     | —     |

The foregoing mixtures (1) to (3) were very good in the kneading steps but extruded bars were rather brittle and were not good in the separation from the die in the case of die-moulding when warm.

Solid soaps from the mixtures (4) to (8) had desirable physical properties but they were not satisfactory in mouldability (not easily moulded and removed) at the temperature desirable for die-moulding and in solubility at a low temperature. This is because the superfatting agent in a free acid state, improves the workability in the mechanical kneading steps and also improves the important physical properties evaluated.

From the foregoing results, it is evident that, in the present invention, as compared with an ordinary commercial toilet soap (mixture 15) of a raw material system in which beef tallow is made a main part and coconut oil less than that is mixed, a solid soap having a practically required solubility without excessive swelling tendencies

can be made without requiring unsaturated fatty acid.

Further, when a saturated fatty acid is added as an excess fat agent in the range prescribed by the present invention, the dried surface is very stable after being dipped in water.

#### Example 2

The dispersibilities for scums in hard water with soaps obtained by the recipes in Example 1 and the effects of the means of the present invention of further improving them are shown in the following test example.

The scum dispersibility was tested according to the method disclosed by N. Schoenfeldt on pages 80 to 83 in the Journal of American Oil Chemists Society, Volume 45, 1958. By varying the conditions as shown, the titration number with 0.01N-HCl was determined and the scum dispersion rate was calculated by the formula:

$$\text{Dispersion rate (\%)} = \frac{\text{Number of ml. of 0.01N-HCl in hard water of } 20^{\circ} \text{ DH}}{\text{Number of ml. of 0.01N-HCl in pure water}} \times 100$$

|    | Kind of Soap<br>(Mixture No.)                                     | Additive and Added Amount<br>(on the Total Soap)   | Scum Dispersion<br>Rate (%) |
|----|---|--|-----------------------------|
| 55 | 1   | —  | 30.5                        |
|    | 2   | —  | 25.4                        |
|    | 3   | —  | 22.6                        |
|    | 6   | —  | 19.7                        |
| 60 | 12  | —  | 39.7                        |
|    | 13  | —  | 55.2                        |
|    | 14  | —  | 45.2                        |
|    | 12  | Polyoxyethylene (2) lauroyl<br>diethanolamide 2%   | 67.5                        |
| 65 | 13  | Polyoxyethylene (2) lauroyl<br>diethanolamide 2%   | 59.3                        |
|    | 12  | Polyoxyethylene (2) lauroyl<br>diethanolamide 4%   | 67.5                        |
| 70 | "   | Polyoxyethylene (5) lauroyl<br>diethanolamide 4%   | 71.4                        |
|    | "   | Polyoxyethylene (10) lauroyl<br>diethanolamide 4%  | 74.2                        |
|    | "   | Polyoxyethylene (5) decanoyl<br>diethanolamide 4%  | 74.5                        |
| 75 | "   | Polyoxyethylene (5) palmitoyl<br>diethanolamide 4% | 75.2                        |
|    | "   | Polyoxyethylene (5) stearoyl<br>diethanolamide 4%  | 73.5                        |
| 80 | Commercial toilet<br>soap of mixed<br>fatty acid titre<br>of 40°C | —  | 31.0                        |
| 85 | Commercial toilet<br>soap of fatty<br>acid titre of<br>44°C       | —  | 25.5                        |

The hard water of 20° DH is based on the German Deutsche Härte scale in which 1° is about 17.9 parts per million as U.S.A. hardness is conventionally expressed. Thus 20° DH is approximately 358 parts per million U.S.A. hardness.

The numbers in parentheses (2), (5), (10) in the table of this example refer to the average number of alkylene oxide units per molecule of the additive specified. These additives exemplify alkanoyl dialkanol amides and polyoxyalkylene alkanoyl dialkanol amides wherein the polyoxyalkylene moieties average from zero to ten alkylene oxide units per molecule and where the alkanoyl moieties have from about 10 to about 18 carbon atoms per molecule. For convenience and reactivity, lower alkylene oxide units are preferred such as ethylene oxide and propylene oxide while lower alkanol moieties are preferred such as ethanol.

#### WHAT WE CLAIM IS:—

1. A process for producing a solid soap, which process comprises saponifying a mixed fatty acid system containing from 60 to 85 percent by weight of a straight chain saturated fatty acid having from 8 to 18 carbon atoms per molecule and from 15 to 40 percent by weight of a branched chain saturated fatty acid having from 10 to 24 carbon atoms per molecule, or their esters, and having a titre of from 35 to 45°C, with about a neutralising amount of a caustic alkali to form a neat soap, and then adding as a superfatting agent from 5 to 20 percent by weight based on the neat soap of a straight chain substantially saturated fatty acid having from 8 to 18 carbon atoms per molecule, there being added to the foregoing mixture or to at least one of the components thereof an inorganic salt in an amount of from 0.3 to 2 percent by weight based on the neat soap.

2. A process as claimed in claim 1, wherein the caustic alkali is a mixture of sodium and potassium hydroxides containing from 10 to 30 mol percent of potassium hydroxide and from 0.5 to 2 percent by weight of salt is employed.

3. A process as claimed in claim 2, wherein the salt is sodium chloride.

4. A process as claimed in claim 1, wherein the mixed fatty acid system contains the acids or their esters with an alkanol, glycol or glycerol, the caustic alkali is an alkali metal or alkaline earth metal hydroxide or an inorganic basic salt thereof, and the inorganic salt is a salt of a strong base and a strong acid.

5. A process as claimed in claim 4, wherein the caustic alkali is an alkali metal or alkaline earth metal hydroxide or carbonate, or a mixture thereof.

6. A process as claimed in claim 4, wherein the caustic alkali is a mixed alkali metal hydroxide containing from 10 to 30 mol percent potassium hydroxide and from 70 to 90 mol percent sodium hydroxide.

7. A process as claimed in any one of the preceding claims, wherein the caustic alkali is provided in an aqueous solution containing an amount of water equal to about 30 percent by weight of the neat soap.

8. A process as claimed in any one of the preceding claims, wherein at least a part of the mixed fatty acid system used is in the form of esters and wherein unsaponifiables are removed following the saponification and prior to the addition of the superfatting agent to the neat soap.

9. A process as claimed in any one of claims 1 to 7, wherein the mixed fatty acid system consists essentially of acids.

10. A process as claimed in any one of the preceding claims, wherein the acids of the mixed fatty acid system are about 80 percent by weight of straight chain acids and about 20 percent by weight of branched chain acids.

11. A process as claimed in any one of the preceding claims, wherein the straight chain saturated fatty acid is caprylic acid, capric acid, lauric acid, myristic acid, palmitic acid, stearic acid, or mixtures thereof.

12. A process as claimed in any one of the preceding claims, wherein the branched chain fatty acid has the formula:



wherein  $R_2$  is an alkyl group having up to four carbon atoms and  $R_1$  is an alkyl group having from 4 to 21 carbon atoms and the total number of carbon atoms of  $R_1$  and  $R_2$  is from 8 to 22.

13. A process as claimed in any one of the preceding claims, wherein a non-ionic surface active agent is added to the soap after saponification.

14. A process as claimed in claim 13, wherein the non-ionic surface active agent is an alkanol amide of a fatty acid having from 8 to 18 carbon atoms per molecule or an alkylene oxide adduct thereof.

15. A process as claimed in claim 14, wherein the alkanol amide is an ethanol-amide.

16. A process as claimed in claim 14 or claim 15, wherein the alkylene oxide is ethylene oxide.

17. A process as claimed in any one of claims 13 to 16, wherein there is employed from 5 to 15 percent by weight of surface active agent based on the neat soap.

18. A process as claimed in claim 1



and substantially as hereinbefore described in Example 1 or Example 2.

19. A solid soap when produced by a process as claimed in any one of the preceding claims.

20. A solid soap when produced by a process as claimed in claim 3 or claim 18.

21. A solid soap comprising soaps of one or more alkali metals and a mixed fatty acid system containing from 60 to 85 percent by weight of a straight chain saturated fatty acid having from 8 to 18 carbon atoms per molecule and from 15 to 40 percent by weight of a branched chain saturated fatty acid having from 10 to 24 carbon atoms per molecule, and having a titre of from 35 to 45°C, from 7 to 30 percent by weight based on the soaps of a straight chain substantially saturated free fatty acid having from 8 to 18 carbon atoms per molecule, and an inorganic salt of a strong base and a strong acid in an amount from 0.4 to 3.0 percent by weight based on the soaps.

22. A soap as claimed in claim 21,

wherein the soaps are soaps of potassium and sodium in proportions from 10 to 30 percent potassium to from 70 to 90 percent sodium, on a mol percent basis.

23. A soap as claimed in claim 21 or claim 22, which also includes from 7 to 22 percent by weight based on the soaps of a non-ionic surface active agent.

24. A soap as claimed in claim 23, wherein the non-ionic surface active agent is as defined in any one of claims 14 to 16.

25. A soap as claimed in any one of claims 21 to 24, wherein the fatty acids of the fatty acid system are as claimed in claim 11 or claim 12.

26. A soap as claimed in claim 21 and substantially as hereinbefore described in Example 1 or Example 2.

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